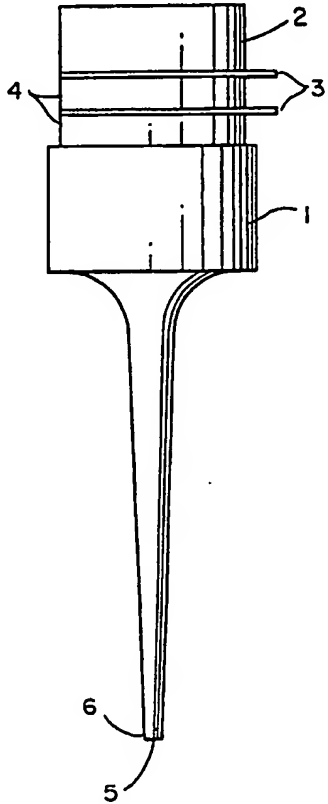




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>5</sup> :  B06B 3/00	A1	(11) International Publication Number: WO 90/14170  (43) International Publication Date: 29 November 1990 (29.11.90)
<p>(21) International Application Number: PCT/US90/02575</p> <p>(22) International Filing Date: 11 May 1990 (11.05.90)</p> <p>(30) Priority data: 351,917 15 May 1989 (15.05.89) US</p> <p>(71) Applicant: E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US).</p> <p>(72) Inventor: STAUNTON, Harold, Francis ; RR2, Box 332A, Avondale, PA 19311 (US).</p> <p>(74) Agents: HAMBY, William, H. et al.; E.I. du Pont de Nemours and Company, Legal Department, 1007 Market Street, Wilmington, DE 19898 (US).</p>		<p>(81) Designated States: AT (European patent), AU, BE (European patent), CH (European patent), DE (European patent)*, DK (European patent), ES (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), NO, RO, SE (European patent), SU.</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: ULTRASONIC PROBE</p> <p>(57) Abstract</p> <p>A piezoelectric transducer having a small-diameter probe (1) whose tip face (5) has a concavity to improve cavitation.</p> 		

### DESIGNATIONS OF "DE"

Until further notice, any designation of "DE" in any international application whose international filing date is prior to October 3, 1990, shall have effect in the territory of the Federal Republic of Germany with the exception of the territory of the former German Democratic Republic.

### *FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MC	Monaco
AU	Australia	FI	Finland	MG	Madagascar
BB	Barbados	FR	France	ML	Mali
BE	Belgium	GA	Gabon	MR	Mauritania
BF	Burkina Fasso	GB	United Kingdom	MW	Malawi
BG	Bulgaria	GR	Greece	NL	Netherlands
BJ	Benin	HU	Hungary	NO	Norway
BR	Brazil	IT	Italy	RO	Romania
CA	Canada	JP	Japan	SD	Sudan
CF	Central African Republic	KP	Democratic People's Republic of Korea	SE	Sweden
CG	Congo	KR	Republic of Korea	SN	Senegal
CH	Switzerland	LI	Liechtenstein	SU	Soviet Union
CM	Cameroon	LK	Sri Lanka	TD	Chad
DE	Germany, Federal Republic of	LU	Luxembourg	TG	Togo
DK	Denmark			US	United States of America

5

## ULTRASONIC PROBE

BACKGROUND OF THE INVENTION

Piezoelectric transducers with probes are well known for their use in ultrasonic processing. Chemists and microbiologists have been using such devices to generate high-intensity cavitation for homogenizing difficult combinations, cell breakage, extraction, particle reduction, cleaning, chemical reaction enhancement, and many other uses.

Such transducers having a needle probe are also known and are principally used for cleaning out small holes.

A need exists for a probe that can be used for the rapid processing of small samples. Such a probe can be used in conjunction with detection chemistry to analyze for the presence of sulfate-reducing bacteria in oil and gas production, cooling and in waste water treatment systems and in pulp and paper production.

When conventional probes having a diameter of less than approximately 0.08 inch (0.2 cm) are employed, it has been found that the cavitation they generate is insufficient to produce the desired effect in many small-volume applications. This is due to predominant edge effects that disperse the sound field at the probe tip, preventing the sound intensity from exceeding the threshold necessary for cavitation.

SUMMARY OF THE INVENTION

I have discovered that the cavitation produced by an ultrasonic probe having a diameter less than 0.08 inch (0.2 cm) can be significantly increased if

the face of the probe tip has a concavity that will focus the sound field, thereby overcoming the edge sound dispersion effects. By concavity it is meant a shallow indentation in the tip face. This concavity can be radially symmetric, e.g., spherical, parabolic, ellipsoidal or conical. It can also be uniaxially symmetric, e.g., grooved with a wedge or curved shape.

Concavities with other symmetries that will focus the sound field and provide the desired cavitation can also be used; however, they would not be preferred as they would be more difficult to fabricate. The preferred concavity is one that is radially-symmetric as it is the easiest to machine, i.e., a simple lathe operation, and they are the most readily finished with a final treatment, i.e., a coating to improve wear resistance.

The shallow concavity in the small-diameter probe tip will generate a relatively extended, stable, high-intensity cavitation zone that readily meets the requirements for sonochemical processing such as rapid cell lysis. This advantage coupled with the small probe diameter means that the apparatus of the invention can be used for small-volume sample processing, can be used with enzymes and other heat sensitive materials as it does not unduly heat the sample, can provide efficient sample circulation for uniform processing, and requires less electrical driving current than larger-diameter probes, thus permitting battery operation. Thus the apparatus of the invention can be readily incorporated into a hand-held, small field unit that can contain its own power supply.

### DESCRIPTION OF THE DRAWINGS

Figure 1 is a side view of a combined  
5 piezoelectric transducer and probe.

Figure 2 is a cross-section of a probe tip  
illustrating a concavity that is radially  
symmetrical, i.e., spherical.

Figures 3 and 4 are further cross-sections of  
10 probe tips having concavities that are conical.

### DETAILED DESCRIPTION OF THE INVENTION

The apparatus of the invention will be  
described with reference to the Figures.

In Figure 1 the probe 1 of a typical  
15 piezoelectric transducer is illustrated. This probe  
can be mounted in a suitable housing (not  
illustrated).

In this embodiment the probe and transducer are  
combined into one piece which is resonant in a single  
20 half-wavelength mode, sometimes referred to as an  
integral-probe transducer. In other embodiments they  
can be in two pieces, a half-wavelength resonant  
transducer and a half-wavelength resonant probe (not  
illustrated). This enables different diameter probe  
25 tips to be employed. If desired more than a one  
half-wavelength probe can be coupled to the  
transducer, e.g. a full-wavelength probe.

The transducer portion of the device shown in  
Figure 1 consists of backpiece 2, electrodes 3,  
30 piezoelectric crystals 4, and a bolt (not  
illustrated) that passes through the backpiece,  
electrodes, and crystals, and threads into the top  
face of probe 1 to compress the crystal/electrode  
sandwich between the probe and the backpiece.  
35 Electrodes 3 communicate between the power source  
(not illustrated) and the piezoelectric crystals 4.

The electrodes can be made of conventional materials, e.g., Be-Cu or nickel and conventional crystals can be employed, e.g., barium titanate or lead zirconate titanate crystals.

The probe 1 can be made of conventional materials, e.g., aluminum or titanium alloys, and in some embodiment it will be desirable to treat or coat the tip face 5 with a wear resistance material, such as chromium oxide, aluminum oxide, an alloy of alumina/titania or similar materials.

In the apparatus of the invention the tip 6 of the probe will have a diameter of less than 0.08 inch (0.2 cm). If the diameter is larger, the tip face 5 need not contain a concavity to produce the desired cavitation.

Figures 2, 3 and 4 all illustrate different embodiments of the shallow concavity on the face of the probe tip. In Figure 2 the concavity is spherical while in 3 and 4 it is conical.

Only a shallow concavity is required in the probe tip to produce the optimum cavitation effect, and, in fact, a deeper feature, i.e. a hole or counterbore, will generate inferior cavitation and cause premature erosion failure within the hole. The preferred concavity is configured with sidewall angles no smaller than 45 degrees to the probe axis, to maximize the vibrational components of the tip surface parallel to the probe axis. The diameter or dimension of the open end of the concavity is preferably a large fraction of the tip diameter or dimension, to maximize the area of focused sound radiation.

Illustrative of the dimensions of the concavity are the following:

In Figure 2, D, the diameter of the probe tip is .050 inch, d, the depth of the concavity, is .010 inch and R, the radius of the sphere is .020.

In Figure 3, D remains the same, while d is .008 inch and  $\alpha$ , the angle is  $60^\circ$ . In Figure 4, D remains the same and d is .012 and  $\alpha$  is  $45^\circ$ .

It should be understood that these dimensions are merely illustrative and many other embodiments are possible, usually depending on the means available to make the concavity.

The concavity in the probe face can be produced by conventional machining operations, e.g., turning, grinding, boring, or electrical-discharge machining.

The apparatus of the invention can be operated in the conventional manner, i.e., frequencies from 20 to 120 kHz depending upon the size and power of the apparatus required. The preferred frequency range for small-sample sonochemical processing is 40 to 70 kHz.

The apparatus of the invention is useful for any application wherein ultrasonic processing is employed. In view of its features it is particularly useful in the rapid treatment of small, heat-sensitive samples. Thus it can be used to lyse sulfate-reducing bacteria for detection by an immunoassay technique. In this use it can be incorporated in a small, field test kit with its own power supply and detection chemistry to analyze biocorrosion problems in oil and gas production.

CLAIMS

5 I claim:

1. In a piezoelectric transducer having a probe for producing cavitation, the improvement comprising the probe tip having a diameter less than 0.08 inch (0.2 cm) and the tip having a concavity on  
10 its face.

2. The probe of Claim 1 wherein the concavity is radially symmetrical.

15 3. The probe of Claim 1 wherein the concavity is uniaxially symmetrical.

4. The probe of Claim 1 wherein the transducer has a frequency range of 20 to 120 kHz.  
20

25

30

35



//

FIG. 1

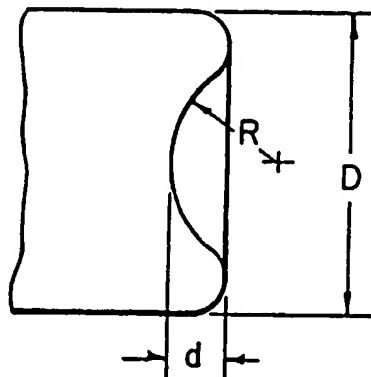
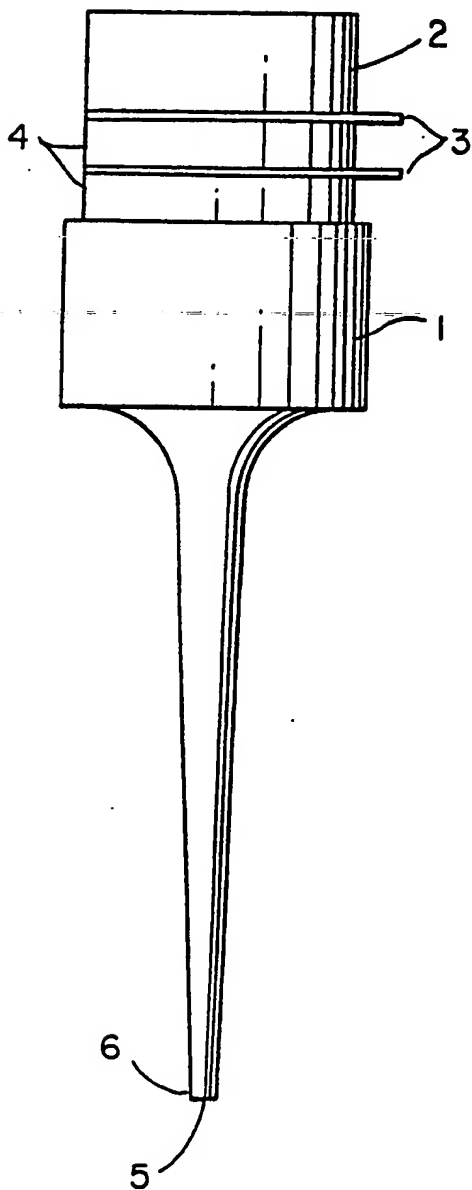


FIG. 2

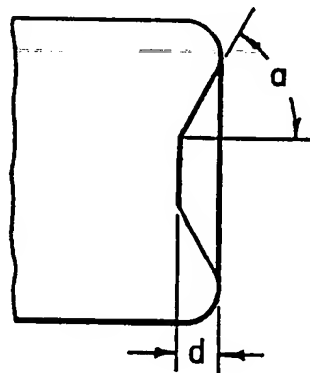


FIG. 3

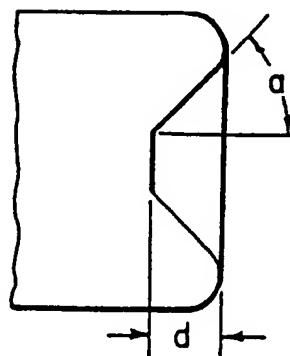


FIG. 4

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US 90/02575

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC <sup>5</sup> : B 06 B 3/00		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
IPC <sup>5</sup>	B 06 B, A 61 F, A 61 B, B 05 B, B 23 K	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>9</sup></b>		
Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	Mass Production, vol. 46, no. 1, January 1970, (London, GB), W.F. Walker: "Ultrasonic welding and brazing", pages 13-19 see figure 7 --	1,2
X	GB, A, 2118045 (GORKOVSKY GOSUDARSTVENNY MEDITSINSKY INSTITUT) 26 October 1983 see page 2, lines 73-90; figure 3 --	1,2
A	US, A, 4589415 (HAAGA) 20 May 1986 see column 8, lines 60-64; figure 6 --	1,2
A	US, A, 4301968 (BERGER et al.) 24 November 1981 see figure 8 --	1,2
./.		
<p><sup>10</sup> Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
2nd August 1990	17. 09. 90	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	Natalie Weinberg	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, " with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	EP, A, 0202101 (TOA NENRYO KOGYO K.K.) 20 November 1986 see page 10, lines 15-19; figure 4 --	1
A	US, A, 2704333 (CALOSI et al.) 15 March 1955 see figures 7,10 -----	1,2

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9002575

SA 36786

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 07/09/90  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB-A- 2118045	26-10-83	CA-A- 1196830	19-11-85
		CH-A- 660846	29-05-87
		DE-A, C 3247124	20-10-83
		FR-A, B 2524798	14-10-83
		JP-A, B, C59000049	05-01-84
		SE-B- 449560	11-05-87
		SE-A- 8303701	29-12-84
		US-A- 4531934	30-07-85
-----			
US-A- 4589415	20-05-86	None	
-----			
US-A- 4301968	24-11-81	US-A- 4153201	08-05-79
		AT-B- 383509	10-07-87
		BE-A- 860540	08-05-78
		CA-A- 1071997	19-02-80
		CA-A- 1090694	02-12-80
		CA-A- 1090695	02-12-80
		CH-A- 627097	31-12-81
		DE-A, C 2749859	10-05-79
		FR-A, B 2386226	27-10-78
		GB-A- 1595716	19-08-81
		GB-A- 1595717	19-08-81
		GB-A- 1595715	19-08-81
		JP-A, B, C53059929	30-05-78
		JP-A- 58092480	01-06-83
		LU-A- 78476	14-03-78
NL-A- 7712249	10-05-78		
SE-B- 434348	23-07-84		
SE-A- 7712563	08-05-78		
-----			
EP-A- 0202101	20-11-86	JP-A- 61259781	18-11-86
		US-A- 4726522	23-02-88
-----			
US-A- 2704333		US-A- 2748298	
-----			